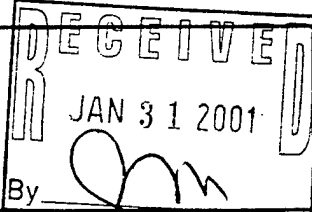


REPORT DOCUMENTATION PAGE			Form Approved OMB NO. 0704-0188	
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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE 01/24/01		3. REPORT TYPE AND DATES COVERED FINAL REPORT - 09/15/95 - 10/15/99
4. TITLE AND SUBTITLE MBE Growth and Properties of GaN, InGaN and GaN/InGaN Quantum Well Structures for Laser Diode Applications			5. FUNDING NUMBERS DAAH04-95-1-0627	
6. AUTHOR(S) J. F. Schetzina				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) North Carolina State University Office of Sponsored Programs Box 7514 Raleigh, NC 27695-7514			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211			10. SPONSORING / MONITORING AGENCY REPORT NUMBER ARO 34836.1 - EL	
11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.				
12a. DISTRIBUTION / AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12 b. DISTRIBUTION CODE	
<div style="text-align: center;">  </div>				
13. ABSTRACT (Maximum 200 words) The program topic was changed to growth of III-V nitrides by MBE by mutual agreement with J. Zavada of ARO. Growth of III-V nitrides by molecular beam epitaxy (MBE) has been studied using rf nitrogen plasma sources. Plasma sources from three different vendors have been tested. All three of the sources have been used to grow high quality GaN. However, the EPI rf source produces an optical emission spectrum that is very rich in the active nitrogen species of 1st-Positive excited nitrogen molecules and nitrogen atoms. GaN growth rates at 800°C of 1µm/hr have been achieved using this source. The MBE-grown GaN films are deposited homoepitaxially on high quality MOVPE-grown GaN/SiC substrates. With the growth conditions for high quality undoped GaN as a baseline, a detailed study of Mg doping for p-type GaN was performed. An acceptor incorporation of $2 \times 10^{19} \text{ cm}^{-3}$ was measured by both CV and SIMS for a doping source temperature of 290°C. However, a faceted 3-dimensional growth mode was observed by RHEED during Mg doping of GaN. Additional studies suggest an interdependence between Mg incorporation and growth surface morphology. Quantum well structures made from the InGaN ternary alloy were grown using a modulated beam MBE method. With this technique, quantum well compositions were controllable grown with visible luminescence ranging from 400nm to 515nm depending on indium mole fraction. Light emitting diode test structures, combin in g Mg p-type doping with InGaN quantum wells, were fabricated and tested.				
14. SUBJECT TERMS III-V nitrides, molecular beam epitaxy (MBE), photoluminescence, electroluminescence			15. NUMBER OF PAGES 24	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OR REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL	

**Final Report for Contract DAAH04-95-1-0627  
9/15/95-10/15/99**

**MBE Growth and Properties of GaN, InGaN and GaN/InGaN Quantum  
Well Structures for Laser Diode Applications**

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**EXECUTIVE SUMMARY**

The program topic has been changed to growth of III-V nitrides by MBE by mutual agreement with J. Zavada of ARO. Growth of III-V nitrides by molecular beam epitaxy (MBE) is being studied using rf nitrogen plasma sources. Plasma sources from three different vendors have been tested. All three of the sources have been used to grow high quality GaN. However, the EPI rf source produces an optical emission spectrum that is very rich in the active nitrogen species of 1st-Positive excited nitrogen molecules and nitrogen atoms. GaN growth rates at 800°C of 1µm/hr have been achieved using this source. The MBE-grown GaN films are deposited homoepitaxially on high quality MOVPE-grown GaN/SiC substrates. With the growth conditions for high quality undoped GaN as a baseline, a detailed study of Mg doping for p-type GaN was performed. An acceptor incorporation of  $2 \times 10^{19} \text{ cm}^{-3}$  was measured by both CV and SIMS for a doping source temperature of 290°C. However, a faceted 3-dimensional growth mode was observed by RHEED during Mg doping of GaN. Additional studies suggest an interdependence between Mg incorporation and growth surface morphology. Quantum well structures made from the InGaN ternary alloy were grown using a modulated beam MBE method. With this technique, quantum well compositions were controllable grown with visible luminescence ranging from 400nm to 515nm depending on indium mole fraction. Light emitting diode test structures, combining Mg p-type doping with InGaN quantum wells, were fabricated and tested.

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## I. INTRODUCTION

Interest in III-V nitrides has greatly increased in recent years due to the commercialization of blue and green light emitting diodes and the demonstrations of blue/violet laser diodes based on nitride heterostructures. These advances have been primarily achieved using metal organic vapor phase epitaxy (MOVPE) as the method of film growth [1-4]. Early efforts to employ MBE for growth of GaN and related materials used ECR plasma sources to generate "active" nitrogen which resulted in very slow film growth rates and poor quality material [5]. More recently, we have shown that high-quality GaN can be prepared by MBE when employing rf nitrogen plasma sources, which produce a greater flux of active nitrogen and therefore, permit MBE film growth at higher temperatures [6]. In addition, high-quality GaN buffer layers, prepared by MOVPE on 6H-SiC substrates at Cree Research, Inc., are being used as substrates for MBE growth. By using a homoepitaxial approach, problems associated with GaN film nucleation on highly lattice-mismatched substrates such as sapphire or SiC have been circumvented and we have been able to focus on the issues associated with the MBE growth process itself [6]. Using this approach, we have studied the doping of GaN, as well as the growth of AlGa<sub>N</sub> and InGa<sub>N</sub> alloys [7].

The doping of GaN with Mg to produce p-type material for device applications is poorly understood at present. Many researchers have reported difficulties in obtaining highly-doped material. In addition, increasing the Mg flux during growth sometimes produces n-type material rather than the expected p-type doping. This phenomenon has been attributed to compensation effects at high Mg incorporation levels. At NCSU, we have studied MBE growth of GaN:Mg systematically for a wide range of Mg oven temperatures, corresponding to different Mg fluxes during MBE film growth. Under optimum growth conditions, SIMS measurements show Mg levels of  $\sim 5 \times 10^{19} \text{ cm}^{-3}$ , corresponding to hole concentrations of about  $5 \times 10^{17} \text{ cm}^{-3}$ .

Special techniques have been developed for the growth of InGaN quantum wells. The growth of high quality InGaN is complicated by thermodynamic limitations: at typical MBE growth temperatures. To overcome these difficulties, we have developed a modulated beam technique which employs alternating layers of (In,Ga)N and (Ga)N, analogous to methods used for the growth of InGaN by MOVPE [8]. The intermittent deposition of a brief GaN layer stabilizes the indium containing layer before droplets can nucleate and results in high quality epitaxy. The resulting GaN/InGaN quantum well structures emit narrow (FWHM ~20-60 nm) room temperature photoluminescence and electroluminescence in the violet/blue/green spectral regions. However, diode structures display relatively poor I-V characteristics, suggesting an additional parallel shorting current path for vertical conduction exists.. Mechanisms which may be responsible for the observed shorting are discussed.

## II. EXPERIMENTAL DETAILS

The GaN films were grown at NCSU using a modified EPI 930 three-chamber MBE system consisting of a main MBE film growth chamber, a second chamber for plasma cleaning of substrates, and a surface analysis chamber with provisions for Auger spectroscopy of substrates and epilayers. The three chambers are interconnected by an ultrahigh vacuum sample transfer system.

The GaN films were grown by MBE on high-quality 2 to 3  $\mu\text{m}$  thick GaN buffer layers prepared by MOVPE on basal plane 6H-SiC substrates. The substrates were cleaned prior to MBE film growth using trichloroethylene, acetone, and methanol followed by plasma-cleaning using a 1:1 hydrogen/helium gas mixture to remove carbon. This was followed by thermal annealing at 800°-1000°C. After cleaning, the substrate surface showed little or no carbon or oxygen contamination. Three different rf plasma sources from different manufacturers were employed to grow undoped GaN. Each of the three rf sources were equipped with pyrolytic boron nitride (pBN) reaction

chambers and exit apertures consisting of 37 small holes (~0.2 mm diameter). Optical emission spectra were using a monochromator equipped with 150 g/mm and 1200 g/mm interchangeable gratings.

A series of p-type GaN:Mg samples was prepared for study by varying the Mg effusion cell temperature from 200°C (lightly doped) to 310°C (heavily doped). Low temperature (4.2-80 K) PL experiments were performed on this set of samples. The modulated beam technique described above was used to prepare a series of GaN/InGaN multiquantum well (MQW) samples. These structures consisted of twenty double layers of GaN/InGaN with double-layer thicknesses ranging from 12 Å to 60 Å.

### III. RESULTS AND DISCUSSION

#### A. Optical Emission Spectra From rf Plasma Sources

Optical emission spectra were obtained from each of the three rf plasma sources and correlated with GaN film growth rates and quality. Fig. 1a and Fig. 1b shows emission spectra obtained for an SVT Associates rf source and an Oxford Applied Research MPD21 rf source, respectively. The sources were operated at the same rf input power and MBE chamber pressures for nitrogen. A number of nitrogen plasma emission peaks are associated with the 1st-positive and 2nd-positive series of neutral nitrogen molecular transitions [7]. The 1st-positive series of molecular nitrogen transitions ( $B^3P_g \leftrightarrow A^3S_u^+$  transitions) appear as five bands of regularly spaced emission peaks in the visible and near-infrared (IR) spectral regions. The strongest 1st-positive emission peak of each band occurs at a wavelengths of 540, 590, 660, 760, and 820 nm respectively. The largest peaks of the 2nd-positive molecular nitrogen series ( $C^3P_u \leftrightarrow B^3P_g$  transitions) occur at 316, 337, 357, 380, and 400 nm. The SVTA source produces a large fraction of 2nd-positive molecular emission peaks in the UV, and the emission appears whitish-violet to the eye. The 2nd-positive emissions correspond to the nitrogen molecules undergoing transitions to a short lived excited state, from which

further decay returns most of the molecules to the tightly-bound ground state (9.85 eV binding energy). Thus, such UV emissions are not expected to indicate significant contribution to the production of "active" nitrogen. Active nitrogen is the term loosely given to nitrogen species that can react to form nitrogen compounds and as such are likely to contribute to MBE growth. In contrast, the Oxford source emission spectrum exhibits diminished 2nd-positive peaks and the emission appears whitish-orange to the eye. Both of these plasma sources produce high quality undoped GaN at comparable growth rates of  $\sim 0.4 \mu\text{m/hr}$  using a GaN substrate temperature of  $800^\circ\text{C}$ . This suggests that the principal reactive nitrogen species responsible for GaN film growth consist of N atoms and 1st-positive  $\text{N}_2$  molecules. 1st-positive transitions leave the nitrogen molecule in a metastable excited state with a binding energy of 3.95 eV from which thermalization may occur at the substrate to form GaN. In this context, growth at higher substrate temperatures will increase the dissociation of the 1st-positive nitrogen molecule and lead to an increased availability of active nitrogen. By using both of the above sources simultaneously, GaN growth rates of  $\sim 0.2 \mu\text{m/hr}$  at growth temperatures of  $900^\circ\text{--}950^\circ\text{C}$  were obtained. In the undoped GaN films, PL emission at 300 K consisted of a single near-band-edge peak at 3.409 eV having a FWHM as narrow as 33 meV. No measurable deep level emission was observed.

The EPI rf nitrogen plasma source employs a Unibulb pBN reaction chamber of original design. This source produces a nitrogen emission spectrum which appears bright orange to the eye and which contains very strong 1st-positive molecular emission peaks and atomic emission lines as shown in Fig. 2. Additionally, the 2nd-positive emission peaks are virtually absent from the spectrum of the EPI source. This emission spectra is what is to be expected when the plasma chamber contains no leakage paths except for the exit apertures. It is well known that, once formed, the nitrogen atom is very long lived, with lifetimes in the order of seconds. This is because collisions between atoms do not result in the formation of nitrogen molecules. To form nitrogen

molecules, two nitrogen atoms in collision must interact with a third body in order to recombine, usually as wall collision. In the EPI plasma chamber, which is designed minimize leakage paths and thus maximize the nitrogen containment time, the plasma becomes richer and richer in atomic nitrogen relative to molecular nitrogen until a steady-state is reached involving wall collisions. Using this source, we have obtained high-quality GaN films at growth rates of more than  $1\text{ }\mu\text{m/hr}$  and growth temperatures up to  $800^\circ\text{C}$ . This is more than a four-fold increase in GaN growth rate compared to the other rf plasma sources investigated. This observed increase in film growth rate confirms that nitrogen atoms and 1st-positive  $\text{N}_2$  molecules are the principal reactive plasma species responsible for GaN film growth. Undoped, n-type, and p-type GaN films have also been successfully grown at high growth rates using the EPI rf plasma source.

#### **B. Magnesium Doping of GaN by MBE**

The optimum Mg cell temperature for p-type doping was found to be  $290^\circ\text{C}$  for GaN growth at  $800^\circ\text{C}$  under N-stable growth conditions. For Ga-stable conditions, little Mg can be incorporated into the GaN layer at this growth temperature. C-V measurements yielded  $N_a - N_d \sim 2 \times 10^{19}\text{ cm}^{-3}$ , corresponding to a hole concentration of about  $5 \times 10^{17}\text{ cm}^{-3}$ . The 300 K PL spectrum from such a layer is shown in Fig. 3.. Note the strong emission at 3.26 eV and weaker emission at 2.94 eV that we associate with the Mg-acceptor, in addition to the band edge peak at 3.41 eV. On the basis of variable temperature PL and reflectance measurements on lightly doped material, we have determined an optical ionization energy for the Mg acceptor of  $224 \pm 4\text{ meV}$  [9].

The Mg-related features grow in magnitude as the Mg oven temperature increases from  $220^\circ$  through  $310^\circ\text{C}$ . However, for a Mg oven temperature of  $>310^\circ\text{C}$  the growth becomes 2-D and the Mg incorporation decreased by several orders of magnitude. C-V measurements confirm that GaN layers grown under these conditions are n-type. We believe that the incoming Mg acts as a surfactant under these

conditions, reflected by the switch to a 2-D growth mode, but is not readily incorporated into the growing layer. It is also known, that under Ga-stabilized 2-D growth, controlled p-type doping of GaN with Mg is not possible at temperatures much above 700 °C because of the high vapor pressure of elemental Mg [9]. However, under N-stable growth conditions, the RHEED pattern becomes spotty, corresponding to a mixture of 2-D and 3-D growth, and Mg incorporation is possible. Using both RHEED and TEM imaging, we find that the surface of the growing GaN:Mg becomes faceted under these growth conditions such that the surface habit becomes the {1-103} planes rather than the expected (0001) basal plane. This suggests that the incorporation of Mg into GaN has a strong planar dependence, due perhaps to surface bonding configurations on different crystallographic planes.

### **C. Growth and Properties of InGaN MQWs**

MBE growth of InGaN alloys is also under study. For quantum well structures emitting visible light, InGaN is essential as the active recombination layer material in double-heterostructure devices such as laser diodes. The growth of high quality InGaN is complicated by thermodynamic limitations: InN is unstable and tends to dissociate at typical MBE growth temperatures of 600°-800°C. Furthermore, the surface energies of InGaN are such that the indium tends to coalesce into metal droplets rather than migrate freely to lattice incorporation sites. The formation of indium droplets results in a low incorporation rate of indium in the growing film and a weak photoluminescence (PL) signal dominated by deep level emission. To overcome these difficulties, we have developed a modulated beam technique, based on ALE and migration enhanced epitaxy, which consists of alternating layers of (In,Ga)N and (Ga)N. The intermittent deposition of a brief GaN layer stabilizes the indium containing layer before droplets can nucleate and results in high quality epitaxy. Factors influencing the InGaN composition include the metal flux ratios, substrate temperature, and the lengths of the beam



modulation periods. Using this technique, GaN/InGaN quantum well structures have been synthesized which emit narrow (FWHM ~20-60 nm) photoluminescence (PL) and electroluminescence (EL) in the violet-to-green spectral regions at room temperature as shown in Figs. 4 and 5.

The InGaN QW structures which emit bright EL at room temperature exhibit SIMS spectra which show excellent control of the InGaN light emitting MQW layer and the GaN:Mg p-doped layer as shown in Fig. 6a. However, the MBE-grown diode structures display relatively poor I-V characteristics, suggesting an additional non-radiative parallel current path exists such that large total currents are required for appreciable light emission. We believe that this shorting mechanism may be related to the columnar nature of present MOVPE/MBE films. Columnar nitride film grown by MOVPE apparently give rise to passivated internal columnar surfaces, whereas MBE growth does not. To check this hypothesis, p-type GaN:Mg layers were deposited onto commercial p-on-n LED structures provided by Cree Research. SIMS spectra for such structures are shown in Fig. 6b. Note that the MBE-grown GaN:Mg surface layer, grown under excess nitrogen plasma flows, shows Mg concentrations up to  $3 \times 10^{20} \text{ cm}^{-3}$ , corresponding to hole concentrations of about  $3 \times 10^{18} \text{ cm}^{-3}$ . Diode structures prepared in this way show excellent diode characteristics (20 mA at 3.6 V), bright light output and no leakage current under reverse bias. These results appear to support the hypothesis that the MBE-grown GaN:Mg layer, which may contain n-type shorting paths at the columnar internal surfaces, is heavily p-type over most of its bulk such that it provides a highly conducting p-type surface layer for the Ni/Au metal contacts .

#### IV. CONCLUSIONS

In summary, we have shown that rf plasma sources produce active nitrogen sufficient for the MBE growth of GaN. Through the comparison of the optical emission spectra under similar operating conditions and the resulting GaN film growth rates, we

have shown that the Unibulb rf source generates the greatest amount of active nitrogen. We have grown a series of Mg doped layers to determine the optimum conditions for p-type doping of GaN. Although we have successfully grown p-type layers at acceptor concentration up to  $\sim 2 \times 10^{19} \text{cm}^{-3}$  in GaN, a 3-D growth mode was observed under the increasing Mg flux necessary for the higher doping levels. Furthermore, there appears to be a surface dependent Mg doping behavior, with higher incorporation observed under 3-D growth conditions relative to 2-D growth conditions. Using a modulated beam MBE approach, high mole fraction InGaN quantum wells, with luminescence controllable from the violet to green spectral region, have been successfully produced. InGaN quantum wells combined with a Mg doped overlayer have been grown and fabricated into EL test structures. These devices emit visible light, however device electrical behavior suggests a parallel conduction path through the p-type layer. We feel that greater understanding of the origin and behavior of this parallel conduction path is a critical factor in optimizing the p-type Mg doping of MBE grown GaN for quantum well optoelectronic device applications.

## **CURRENT AND FUTURE EMPHASIS**

We shall continue to attempt to improve the electrical characteristics of MBE-grown diodes. Two approaches will be investigated during the remaining portion of this contract: (1) We will attempt to passivate the threading dislocations found in the MBE nitride material by using an additional plasma source to generate active hydrogen during MBE growth of nitrides. This approach will enable us to determine if H, missing from the normal MBE environment but present in abundance in the MOCVD case, can act as a passivant for threading dislocations in the MBE-grown nitrides. (2) We will investigate the use of the epitaxial overgrowth technique (ELO) to prepare low dislocation density nitride layers. Nakamura of Nichia Chemical has used this technique for MOCVD growth of nitride laser diode structures that have cw lifetimes of 10,000 hrs.

## ACKNOWLEDGMENTS

We wish to thank Joe Matthews of NCSU for his assistance with substrate preparation and MBE system maintenance. This work is being supported by DARPA and Cree Research internal funds.

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## LIST OF FIGURES

- Figure. 1. Optical emission spectra obtained for SVT Associates and Oxford Instruments rf nitrogen plasma sources for MBE.
- Figure. 2. Optical emission spectrum of EPI rf nitrogen plasma source for MBE.
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- Figure. 6. SIMS spectrum of an MBE grown InGaN MQW diode structure (top) and an MBE grown GaN: Mg layer on MOCVD grown GaN:Mg (bottom).

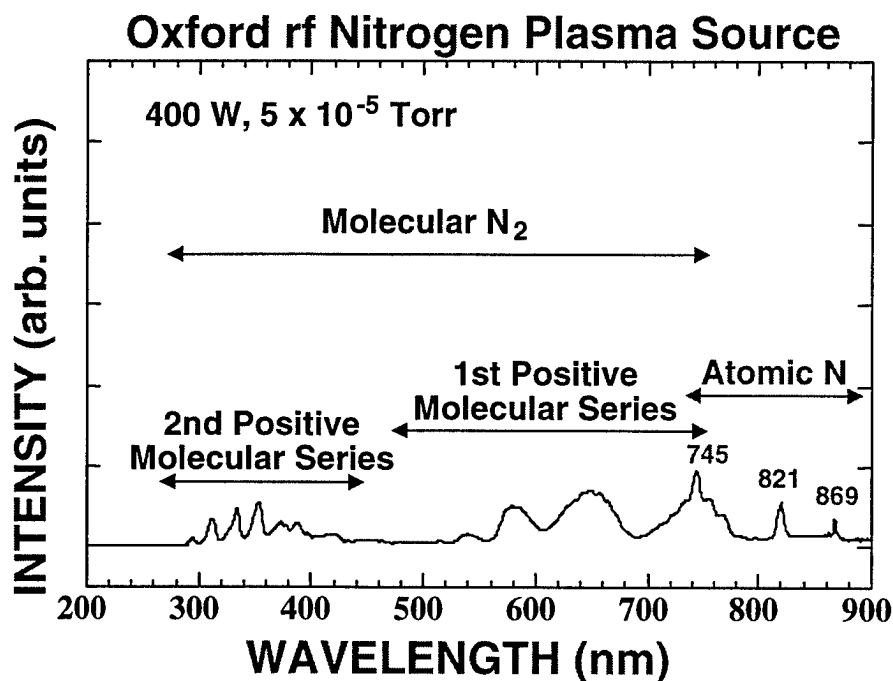
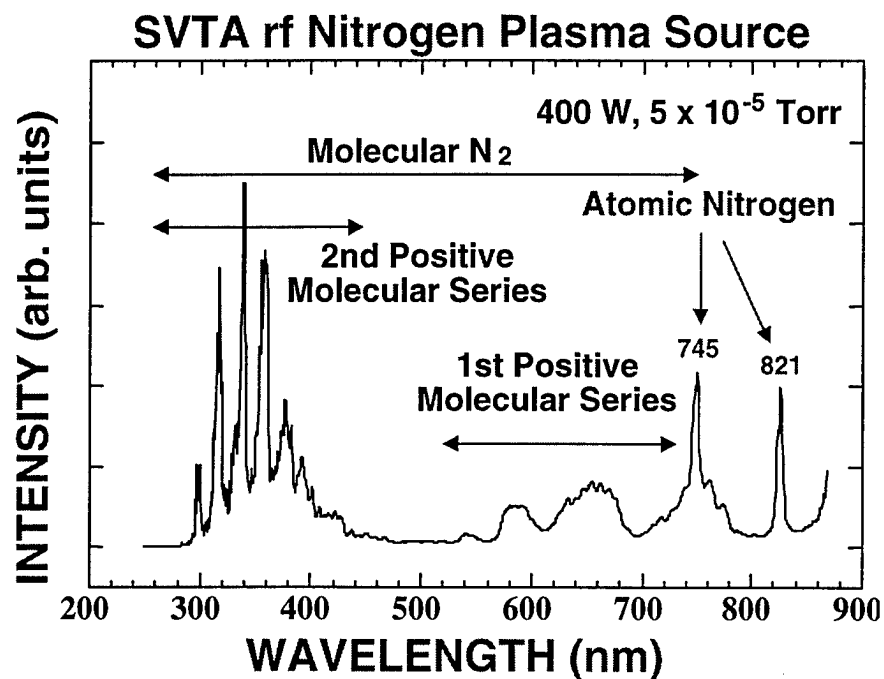


Figure. 1. Optical emission spectra obtained for SVT Associates and Oxford Instruments rf nitrogen plasma sources for MBE.

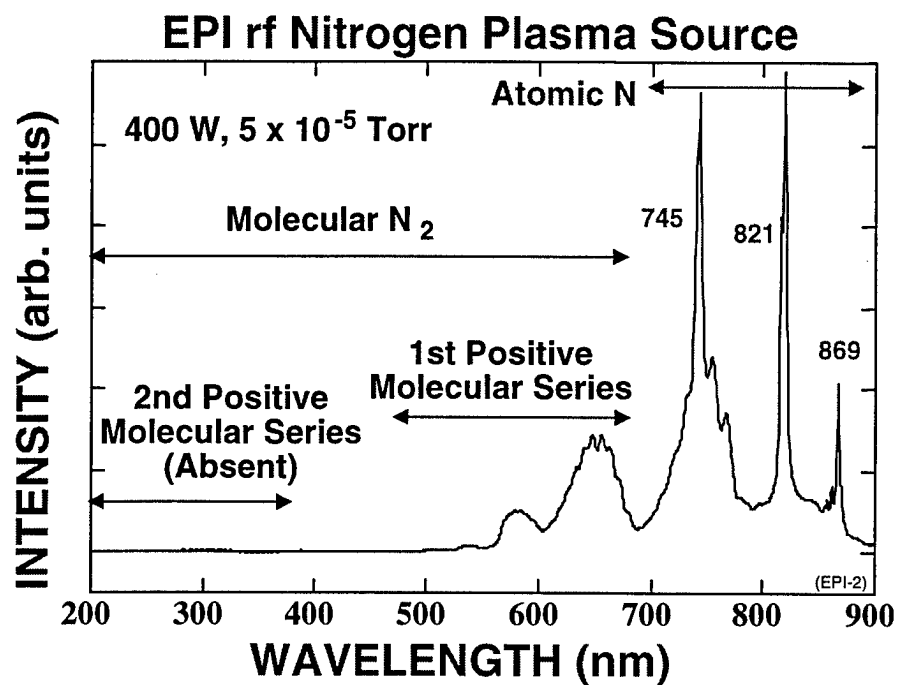


Figure. 2. Optical emission spectrum of EPI rf nitrogen plasma source for MBE.

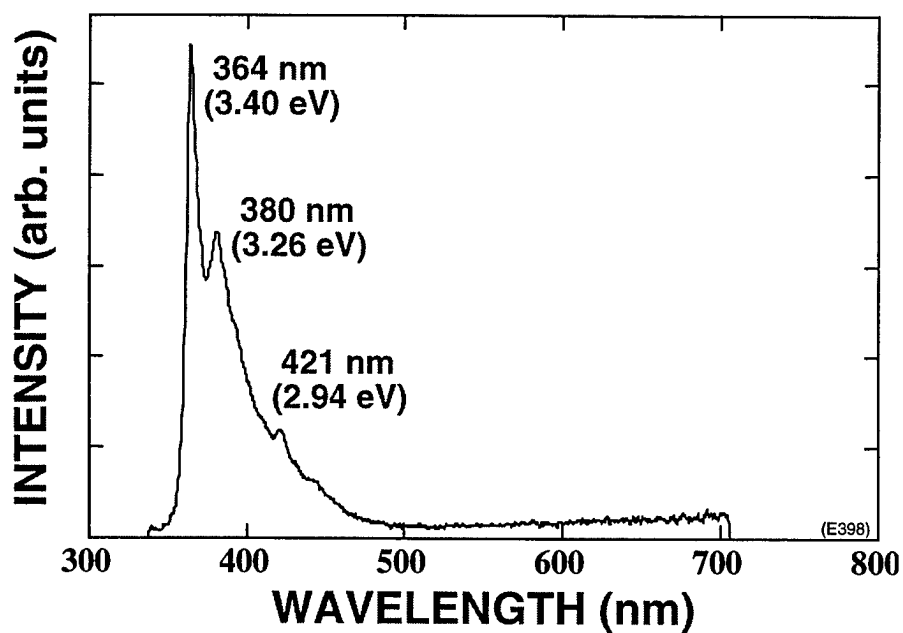


Figure. 3. 300K PL spectrum for GaN:Mg.

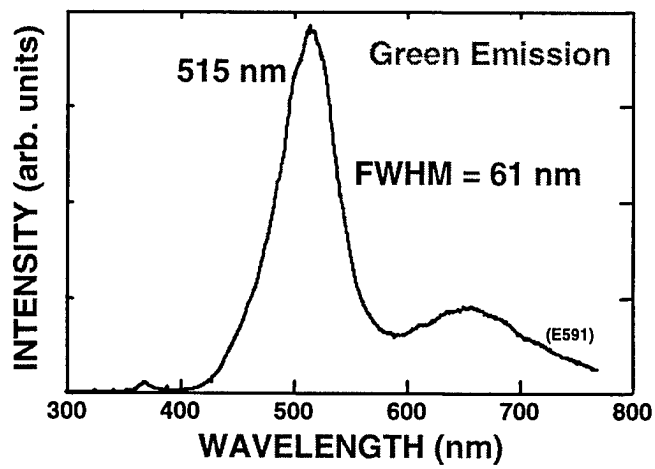
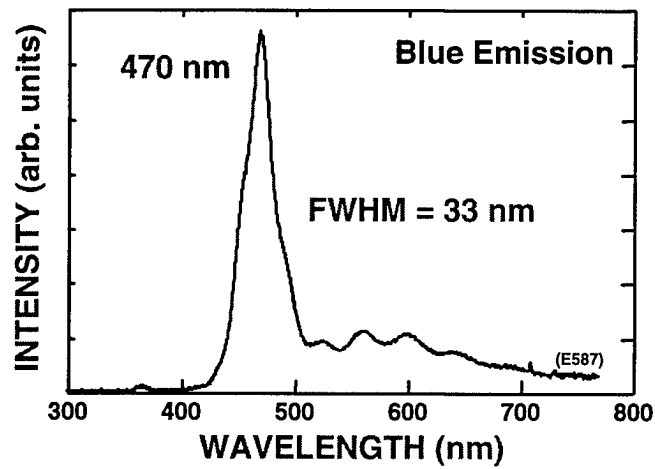
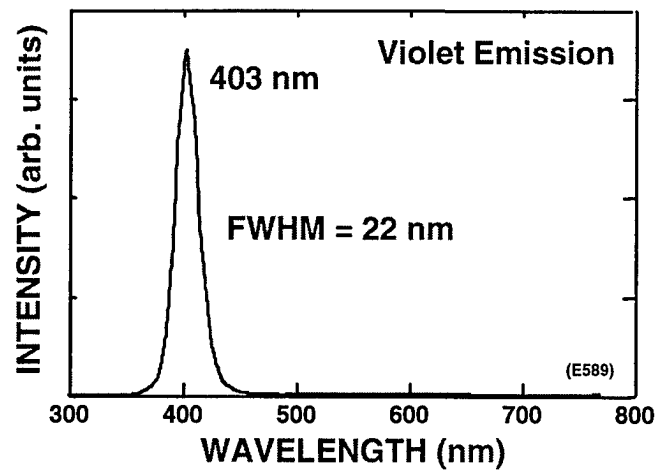


Figure. 4. 300K PL from InGa<sub>N</sub> MQW samples. Violet emission at 403 nm (3.08 eV), blue emission at 470 nm (2.64 eV), and green emission at 515 nm (2.41 eV).

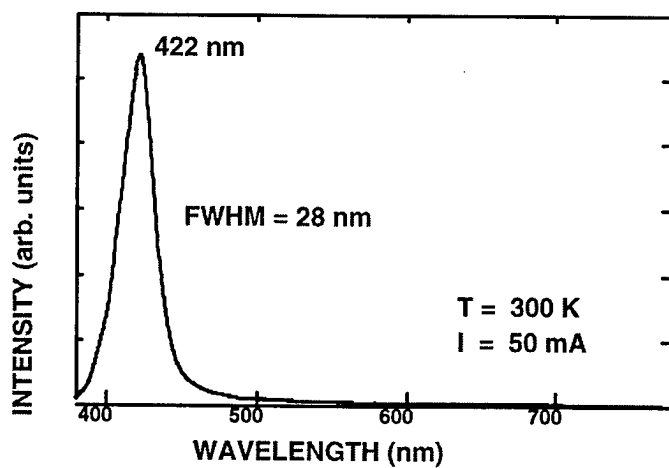
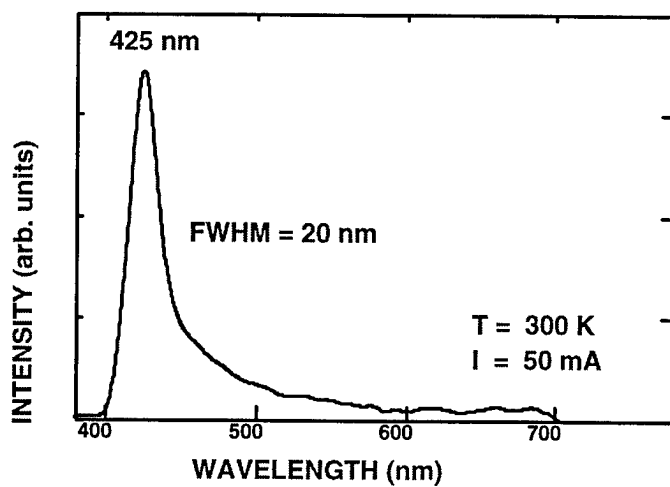
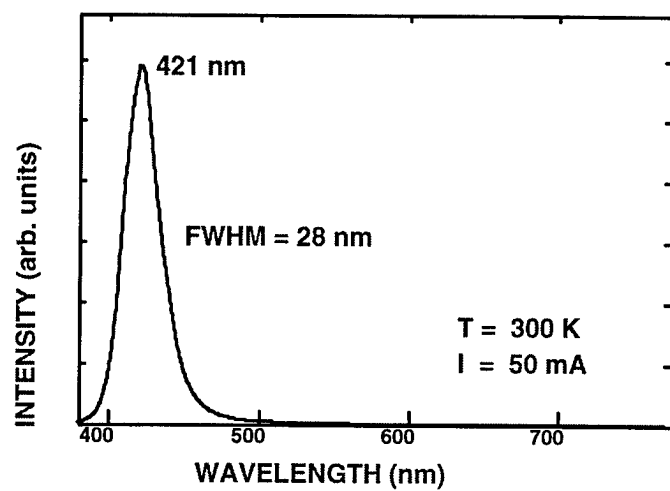


Figure. 5. 300K EL from InGaN MQW diode test structure.



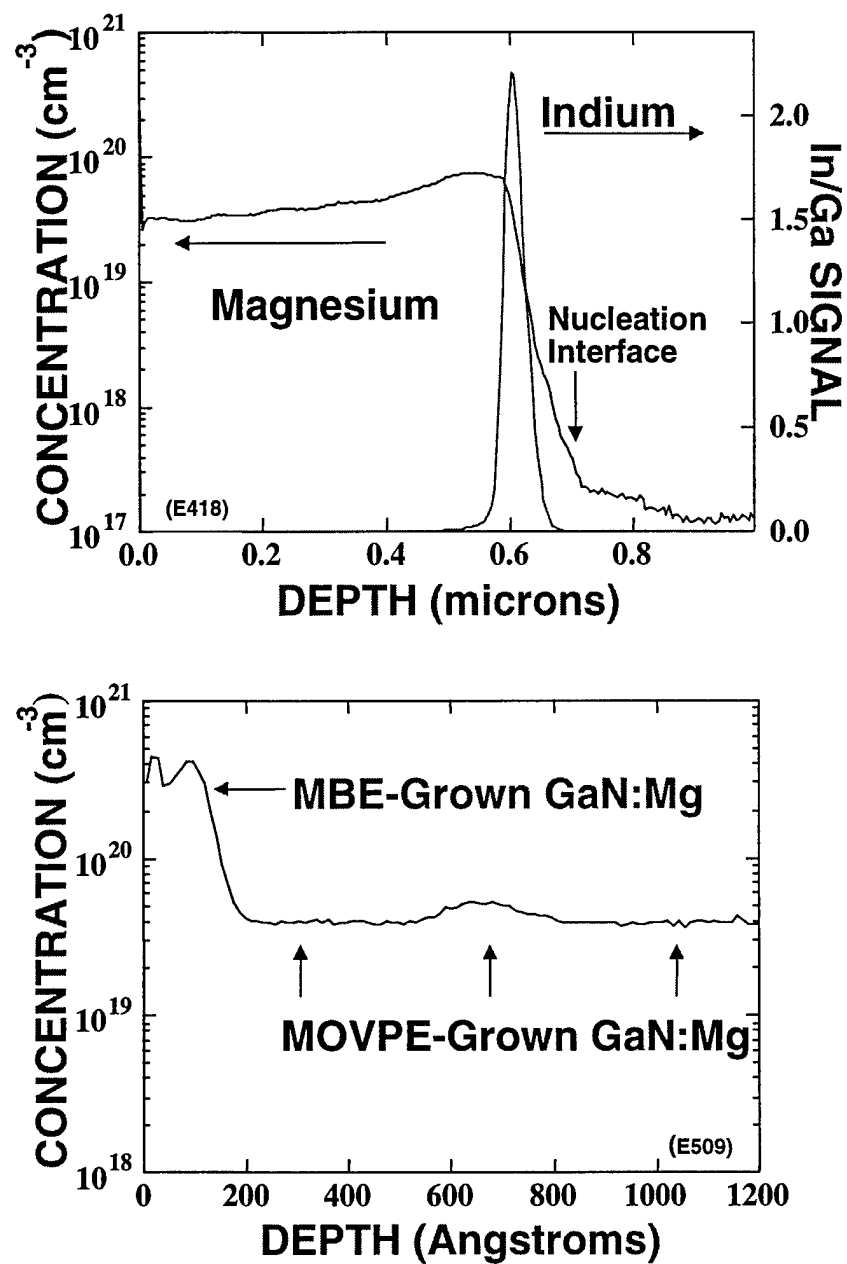


Figure. 6. SIMS spectrum of an MBE grown InGa<sub>N</sub> MQW diode structure (top) and an MBE grown GaN: Mg layer on MOCVD grown GaN: Mg (bottom).

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Post-Doctoral: C. Hughes, J. Yu, Z. Yu

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2. "Blue and Green Light-Emitting Diode Structures Grown by MBE on ZnSe Substrates", D.B. Eason, J. Ren, Z. Yu, W.C. Hughes, J.W. Cook, Jr., J.F. Schetzina, N.A. El-Masry, G. Cantwell and W.C. Harsch, to be published in J. Cryst. Growth, 1995.
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### INVITED PRESENTATIONS

1. "High-Resolution Optical Spectroscopy of ECR and RF Nitrogen Plasma Sources Used for MBE Growth of GaN and Related Alloys", J.F. Schetzina, 1995 Spring MRS Meeting, San Francisco, CA (1995).
2. "Blue/Green Light Emission from II-VI Semiconductor Quantum Well Structures", Physics Colloquium, The Pennsylvania State University, University Park, PA (1995).
3. "Blue/Green Lasers and LEDs on ZnSe Substrates", ARPA Optoelectronics Review, Big Sky, MT (1995).
4. "Blue/Green Light Emitters Based on II-VI Heterostructures on ZnSe Substrates", 1995 International Conference on Solid State Devices and Materials (SSDM'95) Osaka, Japan (1995).
5. "MBE Growth of II-VI Light Emitters on ZnSe", Eagle-Picher Laboratory, Miami, OK (1995).
6. "II-VI LEDs and Laser Diodes on ZnSe", Physics Colloquium, Meijo University, Nagoya, Japan (1995).
7. "Wide Bandgap Semiconductor Heterostructures and Interfaces: Practice and Promise", Third International Conference on Atomic Control of Semiconductor Interfaces (ACSI-3), Raleigh, NC (1995).
8. "Growth And Properties Of III-V Nitride Films, Quantum Well Structures and Integrated Heterostructure Devices, 1995 Fall MRS Meeting, Boston, MA (1995).
9. "Homoepitaxy of Widegap II-VI and III-V Compounds", International Symposium on Blue Laser and Light Emitting Diodes, Chiba, JPN (1996).

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11. "Blue/Green Light Emitting Diodes and Laser Diodes", HBF International Forum on Blue Light Emission for Future Imaging Technology", Tokyo, JPN (1996).
12. "Optoelectronic Applications of Wide Bandgap Semiconductors", 1996 International Surface Science Conference (ISSC), Hong Kong, (1996).
13. "Properties and Applications of Wide Bandgap Semiconductors", Applied Physics Seminar, Cornell University, Ithica NY, (1996).
14. "Wide Bandgap Semiconductor Heterostructures for Optoelectronic Applications", International Conference on Defects and Interfaces in Materials (ICDIM), Wake Forest University, NC (1996).
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16. "MBE Growth of Nitride Heterostructures", Samsung Advanced Institute of Technology, Suwon, KOR (1996).
17. "Optoelectronic Applications of Wide Bandgap Semiconductors", Mitsubishi Chemical Laboratory, Tsukuba, JPN (1996).
18. "Wide Bandgap Semiconductors: Properties, Issues, and Applications", Physics Colloquium, Wake Forest University, Winston-Salem, NC (1996).
19. "Optoelectronic Applications of Wide Bandgap Semiconductors", ECE Seminar, University of California at Santa Barbara (UCSB), Santa Barbara, CA (1996).
20. "MBE Growth and Properties of Wide Bandgap Semiconductors", ARO Symposium on Wide Bandgap Semiconductors, Research Triangle Park, NC (1997).
21. "MBE Growth of GaN", IEEE/LEOS Topical Conference, Montreal, Canada (1997).
22. "MBE Growth of III-V Nitrides", International Conference on Silicon Carbide and Related Materials, Stockholm, Sweden (1997).
23. "MBE Growth of III-V Nitrides for Device Applications", Fall MRS Meeting, Boston, MA (1997)



24. "Device Applications of III-V Nitride Heterostructures", Rockwell Science Center, Thousand Oaks, CA (1998).
25. "Development of Optoelectronic Devices Based on III-V Nitrides", Samsung Advanced Institute of Technology, Suwon, Korea (1998).
26. "MBE versus MOVPE Growth of III-V Nitrides: A Comparison", International Topical Meeting on III-V Nitride Materials and Devices, Beijing, China (1998).
27. "Development of III-V Nitrides for Device Applications", Physics Colloquium, Peking University, Beijing, China (1998).
28. "Optoelectronic Applications of III-V Nitrides", U.S. Army Research Laboratory, Adelphi, MD (1998).
29. "Optoelectronic Applications of Wide Bandgap III-V Nitride Semiconductors", Night Vision and Electrooptic Laboratory, Ft. Belvoir, VA (1998).
30. "UV Detectors and Focal Plane Arrays", U.S. Army Research Laboratory, Adelphi, MD (1998).
31. "UV Digital Camera based on a 32x32 GaN/AlGaIn Photodiode Array", Night Vision and Electrooptic Laboratory, Ft. Belvoir, VA (1999).
32. "Two-Color Imagers Using a UV Focal Plane Array", Fort Monmouth CECOM Headquarters, Fort Monmouth, NJ (1999).
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